TES06 - FTIR Spectroscopy for Polymer Subclass Identification

Scope

This document addresses the performance monitoring QA/QC of the Fourier Transform Infrared (FT-IR) spectrometer system to ensure the instrument is operating within expected parameters; the polystyrene standard listed below must be analyzed each day the instrument is used.

In addition, this document addresses the operating procedures for polymer subclass identification using the Fourier Transform Infrared (FT-IR) spectrometer system. If known and unknown fibers cannot be differentiated utilizing the fiber comparison methods and microspectrophotometric analysis (refer to TES04 – Forensic Fiber Examination), the fibers may be analyzed utilizing FT-IR. The analysis involves two steps: a comparison of the known and/or questioned fibers to one another, and an identification of the polymeric material. The technique is limited to the analysis of manufactured fibers. Natural fibers are not typically analyzed utilizing FT-IR.

Safety Precautions

Universal precautions will be followed. No specific hazards are associated with the microscopic techniques performed.

Materials Required

- Thermo Nicolet 6700 FT-IR (or equivalent)
- Liquid nitrogen
- Dewar flask (supplied with instrument)
- Polystyrene standard – 1.5 mil (38 micron) matte-finish film mounted on a card supplied with the microscope
- Slide containing a metal disk with a 100 micron pinhole, an open hole approximately 11 mm in diameter, and a 14 mm diameter gold disk
- Diamond Compression Cell
- Xylene or Xylene substitute
- Glass microscope slides and cover slips
- Stereobinocular microscope, magnification range from 0.5x to at least 40x
- Carborundum pen, forceps, scalpel

Standards and Controls

- **Daily Performance Standard**
  The 1.5 mil polystyrene film is analyzed as the performance standard to assess daily operating performance, wavenumber assignment, and continued integrity of the system. The polystyrene standards used for this procedure require no preparation. Although the polystyrene standards do not expire it is recommended that they be replaced every two years due to degradation of the standard or if they begin to show signs of wear or if results have drifted.

- **Monthly Performance Standards**
  The 1.5 mil polystyrene film is used to evaluate the microscope accessory. The polystyrene standards used for this procedure require no preparation. Although the polystyrene standards do not expire it is recommended that they be replaced every two years due to degradation of the standard or if they begin to show signs of wear or if results have drifted.

Procedure

Quality Control Daily Calibration

1. Cool the detector with liquid Nitrogen. The Nexus 6700 holds between 500mL and 700mL. The liquid Nitrogen is available from the chemistry unit.
2. Open the OMNIC software program on the computer.
3. The experiment tab is set to default, change this to Scope %T.
4. Set the condenser to the "0" setting.
5. Place the 1.5 mil thick polystyrene standard on the stage.
6. Lower plastic sheath (located around objective) over sample.
7. Focus the sample.
8. Check alignment of the reflex aperture.
    a. Turn the microscope to reflectance mode.
    b. Turn the transmission and reflectance illuminator controls down.
    c. Turn the reflex aperture illuminator dial all the way up.
    d. With the condenser centering screws move the top collection square off of center if necessary. You should now be able to see the bottom collection square of the reflex aperture.
    e. Focus the bottom collection square using the focusing knob.
    f. Increase the transmission illuminator intensity.
    g. Focus the edges of the top collection square using the collection square focusing knob.
    h. Align the collection squares using the condenser centering screws to center the reflex aperture.
9. Place the scope back into transmittance mode.
10. In the computer software select "Collect Sample".
    a. Enter the sample name (for the standard type in polystyrene and the date).
11. When the scan is finished it will automatically prompt you to prepare for a background scan. Remove the polystyrene film and select "OK".
12. Once the scan is done, change the collection and processing information (use the green information button next to the scroll down window).
    a. Under the comments section, type in spectra data:
        Nexus 6700
        Scope %T
        1.5 mil Film

   b. Select "OK".
13. To determine the peak's frequency go to "Analyze", "Find Peaks". Move the cut-off line up and down until you have all of the necessary peak frequencies. Examine the polystyrene standard spectrum for peak wave numbers at 3025 cm^{-1}, 1601 cm^{-1}, 1028 cm^{-1}, and 906 cm^{-1}.
14. To save the spectrum go to "File", "Save as" – Open the correct folder and select the set file name to title button, then hit "Save".
15. To print the spectrum:
    a. Set the template by selecting "Report", "Template" and select the one you want (FBI initials for a sample or standard, one of the library options if you are printing a spectra that has been stored in a library).
    b. Preview and print the report by selecting "Report", "Preview", and "Print Report". A box will open; type your initials and select "OK".
16. Fill out the daily check in the QC log book and add a copy of the polystyrene spectra to the FBI and MPD QC log books. The polystyrene spectrum is acceptable if the following four peaks are within a +/- 4 cm^{-1} (wavenumber) window of the expected value. If the values lie outside the specified range, re-align the microscope and re-analyze the polystyrene. If the results are still failing, contact the TEU equipment manager.

Expected value	Acceptable range
3025 cm^{-1}	3021 to 3029 cm^{-1}
1601 cm^{-1}	1597 to 1605 cm^{-1}
1028 cm^{-1}	1024 to 1032 cm^{-1}
906 cm^{-1}	902 to 910 cm^{-1}

Monthly QA/QC of FT-IR Microscope Accessory (performed by FBI personnel)

1. Cool the detector with liquid Nitrogen. The Nexus 6700 holds between 500mL and 700mL. The liquid Nitrogen is available from the chemistry unit.
2. Open the OMNIC software program on the computer.
3. The experiment tab is set to default, change this to Scope %T.
4. Set the condenser setting to "0".
5. Align the reflex aperture collection windows and focus the microscope using the 100 micron pinhole slide.
6. Monitor the interferogram signal under a gain setting of 1.0 on the continuum.
7. Record the peak-to-peak voltage of the interferogram in the instrument logbook.
8. Retain a copy in the MPD logbook.

Sample Preparation

1. Cool the detector with liquid Nitrogen. The Nexus 6700 holds between 500mL and 700mL. The liquid Nitrogen is available from the chemistry unit.
2. Remove the fiber from the slide.
    a. Punch a hole in the coverslip with a carborundum pen
    b. With a carborundum pen, chip away the glass
    c. Apply a solvent (Xylene) to the slide

   d. With tweezers carefully extract the fiber. If it is a long fiber only remove part of it by cutting a portion out from the slide, leaving a portion still mounted on the slide. If the fiber is short remove the entire fiber.
3. Place the extracted fiber on a clean glass microscope slide in a drop of Xylene.
4. Cut the fiber into pieces and remount a portion of the fiber in Permount
5. Wash the fiber to be analyzed with the FTIR in xylene three times and cut into two small sections
6. Place the two sections of the fiber in an air mount with a coverslip taped down.
7. Prepare the diamond cell:
   a. Remove the screw-top cap, both diamond anvils, and the O-ring from the holder.
   b. Clean the surface of the diamond anvils with Xylene/Xylene substitute.
   c. Place one diamond anvil under stereomicroscope with the diamond face up.
8. With tweezers, carefully remove a small section of the fiber from the air mount and place it on the middle of the face of the diamond anvil.
9. Gently position O-ring and top diamond anvil on top of the bottom anvil, making sure the fiber is in the field of view.
10. Transfer the diamond cell to the holder with the tweezers.
11. Tighten the screw top almost as tight as it will go. You can check the fiber thickness under the stereoscope.
12. Remove the screw-top, top diamond plate, and O-ring, leaving the fiber on the bottom diamond plate in the holder. If the fiber is stuck to the top diamond plate, remove the bottom plate and place the top plate and compressed fiber in its place. The sample is ready to run.

Sample Analysis

1. Open a new sampling window by clearing the current window or going to "Window", "New Window".
2. Place prepared sample (on one diamond cell) firmly in the diamond cell holder on the stage.
3. Change the condenser setting to "1".
4. Center the specimen and lower the plastic objective sheath over the sample.
   Focus on the sample and re-align the reflex aperture so that the long side of the reflex aperture is parallel with the sample. The shape and angle of the collection windows can be adjusted by using the reflex aperture controls. The controls allow you to rotate the aperture and adjust the width and height of the reflex aperture.
5. With the stage controls, move the fiber so that the collection windows are placed over the fiber. The collection windows should be smaller than the fiber and completely fit inside the edges of the sample.
6. Verify that the instrument is in transmission mode.
7. With the computer software select the "Collect Sample" button. Enter the title (Case number, item number, etc.) and select "OK".
8. The computer will prompt the operator to prepare the sample for background collection. Move the collection windows off of the fiber, but still focused on the diamond cell, lower the objective sheath, and select "OK". A background will automatically be run.

9. Once the scan is done, enter the collection and processing information (use the green information button next to the scroll down window).
    a. Under the comments section, type in spectrum data:
        Nexus 6700
        Scope %T
        Flattened between two diamond cells
        Sample taken on one diamond
    b. select "OK"
10. To save the spectrum go to "File", "Save as", Open the correct folder and select the "Set file name to title" button, then hit "Save".
11. To print the spectrum
    a. Set the template by selecting "Report", "Template" and select the one you want (select the FBI initials template for a sample or standard or chose a library template if you are printing a spectrum that has been stored in a library).
    b. Preview and print the report by selecting "Report", "Preview", and "Print Report". A box will open; type your initials and select "OK".
12. Repeat steps 1 through 11 for additional samples.

Sample Identification

A polymeric identification will be attempted using the instrumental reference libraries that are available.

1. Set up the library you wish to search against by going to "Analyze", "Library Setup". Select the desired library (Preferably the FBI Fiber Library), and hit "OK".
2. Search the library by going to "Analyze", "Search". A list of possible spectra will appear.
3. Select the spectra you want to examine (to select multiple spectra hold down the Ctrl key as you click on each spectrum), and copy them (Ctrl + C).
4. Open up the window the sample spectrum is stored in and paste (Ctrl + V) the library spectra into the window.
5. Compare the spectra by highlighting the area of interest to be compared. Move the enlarged peaks up and down (by clicking and dragging on each spectrum) so as to compare the peak positions.
6. To view the entire spectra again, double click below the spectra and then select "View", then "Full scale".
7. Print reports for the sample, the library match, and the sample and library match together (optional).
    a. Set the template by selecting "Report", "Template" and select the one you want (select the FBI initials template for a sample or standard or chose a library template if you are printing a spectrum that has been stored in a library).
    b. Preview and print the report by selecting "Report", "Preview", and "Print Report". A box will open; type your initials and select "OK".

Sample Comparison

Two samples can be compared utilizing the FT-IR.

1. Open up the spectra files for the desired samples.
2. Compare the spectra. Highlight the area of interest on the spectra and moving them up and down (by clicking and dragging on the spectrum) to focus on the desired peaks.
3. To view the entire spectrum, select "View", "Full Scale".
4. Print reports for the sample, the library match, and the sample and library match together.

Shutting Down the Instrument and Computer:
When you are finished for the day close out of the OMNIC software program and turn down the light intensities of the three reflex aperture knobs. Do NOT turn the bulbs all the way off, shut off the instrument, or shut off the computer.

Acceptance Criteria
Two samples can be associated utilizing FT-IR when no unexplainable differences are found between their spectra. A copy of the spectra used in a fiber association will be included in the case notes.

1.4 Instrument Repair and/or Maintenance

If the instrument has undergone repair and/or maintenance, or the instrument goes outside the control of the MPD Laboratory, calibration verification must be performed before being used in casework. The procedure for calibration verification is described in the Daily Calibration Section of the Quality Control Procedure.

If following repair and/or maintenance the calibration and/or calibration verification is performed by an external source (e.g., FBI TEU), the MPD Trace Evidence Unit must verify and document who performed the verification and if the results are acceptable. In addition, a daily performance standard must be tested by the MPD Trace Evidence Unit prior to use.

Limitations

Only properly trained personnel shall perform the duties involved in the operation, maintenance or troubleshooting of this instrument.

The technique is limited to the analysis of manufactured fibers. Natural fibers are not typically analyzed utilizing FT-IR.

Comments

Not applicable.

Documentation

The following worksheet(s) shall be generated and managed:

Casework Documentation

FTIR user QC log

References

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